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Specification as originally filed, with Application for Patent Serial No: 2,409,580,
on October 22, 2002, by DUPONT CANADA INC., assignee of Nicholas A. Farkas,
Colin Tudor and Amitkumar N. Dharia, for "Scratch and Mar Resistant Soft Ethylene
Elastomer Compounds".

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O P I C  C I P O

Abstract

A soft ethylene elastomer composition with improved scratch and mar resistance properties is provided wherein the composition comprises a soft ethylene elastomer component and an effective amount of mica and compatibilizer to impart improved scratch and mar resistance properties to the composition while not decreasing the softness of the composition.

SCRATCH AND MAR RESISTANT SOFT ETHYLENE ELASTOMER COMPOUNDS

Field of the Invention

The invention relates to the field of soft ethylene elastomer compounds and in particular to soft ethylene elastomer compounds exhibiting improved scratch and mar resistant properties.

Background to the Invention

The manufacture of exterior automotive parts has traditionally involved hard ethylene elastomer compounds, which provide good impact, scratch and mar resistance properties to the automobile exterior. These ethylene elastomers comprise a majority of polypropylene, a lesser amount of a rubber toughener (EPDM or EPR rubber) and a filler such as talc.

More recently compounds have been developed with added softness. These compounds called "soft ethylene elastomer compounds" have a polyethylene plastomer as the major component of the composition and polypropylene as a minor component. The addition of a small amount of organic peroxide, optionally with a co-agent, changes the melt rheology characteristics of the compound thereby improving thermal resistance and processing characteristics of the compound and providing a soft characteristic to the compound.

An example of a soft ethylene elastomer compound is disclosed in International Patent Application No. WO 98/32795 to Heck et al. published July 30, 1998, which is incorporated herein by reference. WO 98/32795 discloses a rheology-modified, substantially gel-free ethylene elastomer composition comprising an ethylene alpha-olefin (EAO) polymer or EAO polymer blend and at least one high melting polymer selected from polypropylene homopolymers and propylene/ethylene (P/E) copolymers. The composition has at least three of the following four characteristics: a shear thinning index (STI) of at least 20, a melt strength (MS) of at least 1.5 times that of the composition without rheology modification, a solidification temperature (ST) of at least

10°C greater than that of the composition without rheology modification, and an upper service temperature (UST) limit of at least 10°C greater than that of the composition without rheology modification.

The EAO polymer or EAO polymer blend of the ethylene elastomer is present in an amount of 50-90 % by weight of this component, preferably 65-85 % by weight while the high melting point polymer is preferably present in an amount of 50-10% by weight, more preferably 35-15% by weight.

WO 98/32795 also discloses a process for preparing a rheology-modified, substantially gel-free thermoplastic elastomer composition, which includes treatment with an organic peroxide, and manufacture of articles using these compositions.

For automotive interior and other applications that require scratch and mar resistance, soft ethylene elastomer compositions have been deficient due to their soft polymeric nature. While attempts have been made to add fillers such as talc, wollastonite, calcium carbonate, or other additives such as low molecular weight slip additives, to ethylene elastomer compounds to improve scratch and mar resistance, all have provided less than optimal results. In particular, significant issues have persisted due to one or more of the following: (a) higher gloss after filler or additive addition, (b) scratch whitening because of inadequate adhesion between the filler and polymer phase, (c) poor thermoformability of sheets made from said compounds, (d) adverse effects of filler on compound physical or thermal properties.

As a result there still remains a need to provide a soft ethylene elastomer composition with improved scratch and mar resistance properties.

Summary of the Invention

According to one aspect of the invention, there is provided a composition comprising a soft ethylene elastomer component and an effective amount of mica and compatibilizer

to impart improved scratch and mar resistance properties to the composition while not decreasing the softness of the composition.

Preferred embodiments of the present invention also include a process for preparing the composition and an article of manufacture which has at least one component fabricated from the composition.

Detailed Description of the Invention

In a preferred embodiment, the ethylene elastomer component used in the present invention is a rheology-modified ethylene elastomer composition comprising from about 50 to about 90% by weight of elastomeric ethylene polymers or EAO polymer blend and from about 10 to about 50% by weight of a high melting polymer, based on the total ethylene elastomer composition.

Elastomeric ethylene polymers that are suitable for use in the ethylene elastomer composition include interpolymers and diene modified interpolymers. Illustrative polymers include ethylene/propylene (EP) copolymers, ethylene/butylene (EB) copolymers, ethylene/octene (EO) copolymers, ethylene/alpha-olefin/diene modified (EAODM) interpolymers and ethylene/propylene/diene modified (EPDM) interpolymers. More specific examples include ultra low linear density polyethylene (ULDPE) such as Attane™ manufactured by The Dow Chemical Company, homogeneously branched linear EAO copolymers such as Tafmer™ manufactured by Mitsui PetroChemicals Company Limited and Exact™ manufactured by Exxon Chemical Company, and homogeneously branched substantially linear EAO polymers such as the Affinity™ polymers available from The Dow Chemical Company and Engage® polymers available from DuPont Dow Elastomers L.L.C.

Preferred EAO polymer blends include homogeneously branched linear and substantially linear ethylene copolymers with a density measured in accordance with ASTM D-792 of 0.85-0.92 g/cc, especially 0.85- 0.90 g/cc and a melt index or I_2 (measured in accordance with ASTM D-1238 (190°C/2.16 kg weight) of 0.01-30 preferably 0.05-10 g/10 min.

These substantially linear ethylene copolymers or interpolymers (also known as "SLEPs") are especially preferred.

In addition, the various functionalized ethylene copolymers such as ethylene vinyl acetate (EVA), which contain from 0.5-50 wt % units derived from vinyl acetate are also suitable. When using an EVA polymer, those that have an I_2 of from 0.01-500, preferably 0.05-50 g/10 min are preferred.

Preferred SLEPs include Engage® polyolefin elastomers and other polymers produced by The Dow Chemical Company and DuPont Dow Elastomers L.L.C.

The high melting polymer useful in the ethylene elastomer component is preferably a homopolymer of propylene, a copolymer of propylene with an α -olefin such as ethylene, 1-butene, 1-hexene or 4-methyl-1-pentene. The polymer may also be a blend of a homopolymer and a copolymer, a nucleated homopolymer or a nucleated copolymer. It may further be a nucleated blend of a homopolymer and a copolymer. The α -olefin is preferably ethylene. The copolymer may be a random copolymer or a block copolymer or a blend of a random copolymer and a block copolymer. The high melting polymer is preferably selected from polypropylene homopolymers and polyethylene copolymers with a melt flow rate (MFR) of (230°C and 2. 16 kg weight) of 0.3-60 g/10 min, preferably 0.8-40 g/10 min and more preferably 1-35 g/10 min.

A more detailed description of the preferred elastomeric ethylene polymer or EAO polymer blend and high melting polymer is provided in WO 98/32795, which is herein incorporated by reference.

The ethylene elastomer component is preferably a rheology-modified, substantially gel-free composition treated with a suitable organic peroxide in the manner described in WO 98/32795. Such suitable organic peroxides used for the purposes of this invention have a half-life of at least one hour at 120° C. Illustrative peroxides include: a series of vulcanizing and polymerization agents that contain α , α' -bis(t-butylperoxy)-diisopropylbenzene and are available from Geo Specialty Chemicals under the trade-

mark Vulcup™, a series of agents that contain dicumyl peroxide are available from Geo Specialty Chemicals under the trade-mark Di-cup™, Luperox™ peroxides made by Atofina, North America, or Trigonox™ organic peroxides made by Moury Chemical Company.

Other suitable peroxides include dicymyl peroxide, 2,5-dimethyl-2,5-di-(t-butyl peroxy)hexane, di-t- butylperoxide, 2,5-di(t-amyl peroxy)-2,5-dimethylhexane, 2,5-di-(t-butylperoxy)- 2,5-diphenylhexane, bis(alpha-methylbenzyl)peroxide, benzoyl peroxide, t-butyl perbenzoate and bis(t-butylperoxy)-diisopropylbenzene.

A more detailed description of the properties of the rheology-modified ethylene elastomer composition is provided in WO 98/32795, which is herein incorporated by reference.

The peroxide may also be used with a suitable co-agent such as Type I polar co-agents, which are have a relatively low molecular weight. An example of this type of co-agent is trimethylopropane trimethacrylate (TMPTMA) sold by Sartomer under the name SR-350. Type II low polarity co-agent may also be useful such as 1,2-polybutadiene sold by Sartomer under the trade-mark Ricon®. The addition of co-agent may improve the properties of the rheology-modified ethylene elastomer compound, or reduce the amount of peroxide needed to effect the same level of rheology modification.

The preferred ratio of peroxide to co-agent used in this invention is in the range of about 1:1 to about 1:4 peroxide:co-agent, but may vary depending on the particular application.

The soft ethylene elastomer component is combined with an effective amount of mica and compatibilizer to provide a composition with improved scratch and mar resistant properties.

Preferably, the mica is present in an amount of from about 1 to about 20 % by weight of the total composition, preferably between from about 2 to about 15% by weight, more preferably from about 3 to about 12% by weight, and further more preferably from about 4 to about 10% by weight.

The mica is preferably a wet ground mica of 325 mesh. Preferably a muscovite mica is used or alternatively a phlogophite mica.

The addition of the mica and compatibilizer into the composition of the present invention may be by direct addition to the ethylene elastomer component during compound manufacture or preferably by addition through a pre-made mica masterbatch. If a mica masterbatch is used, it is preferably comprised of mica, compatibilizer and base resin in which the compatibilizer comprises from about 1 to about 20% of the total masterbatch composition, while the mica comprises from about 10 to about 90% of the total masterbatch composition. Preferably, the mica masterbatch comprises from about 5 to about 15% compatibilizer and from about 30 to about 60% mica.

The compatibilizer is preferably a functionalised polyolefin comprising maleic anhydride grafted Engage® or equivalent metallocene polyethylene plastomer, maleic anhydride grafted ethylene propylene copolymer, maleic anhydride grafted ethylene propylene diene monomer, maleic anhydride grafted linear low density polyethylene, maleic anhydride grafted very low density polyethylene or maleic anhydride grafted polypropylene. The compatibilizer acts as a coupling agent in so far as it helps to increase the adhesion in the mica to the soft ethylene elastomer component. The compatibilizer has a polar portion that binds to the mica and a non-polar portion is compatible with the soft ethylene elastomer component to ensure effective adhesion between the mica and the soft ethylene elastomer component.

In a preferred composition of the present invention, the compatibilizer comprises from about 0.1 to about 7%, preferably from about 0.25 to about 5% by weight of the total composition.

The base resin in the masterbatch is preferably a carrier resin such as an Engage® polyolefin plastomer, linear low density polyethylene, very low density polyethylene or ethylene propylene copolymer. Where the base resin in the masterbatch is a functionalised resin such as ethylene vinyl acetate, or a copolymer of ethylene and methylacrylic acid or acrylic acid, the functionalised resin may behave in part as a

compatibilizer. The base resin in the masterbatch is typically compatible with the elastomeric ethylene polymer of the composition.

The masterbatch may also comprise additional additives or specialty additives without deviating from the invention as claimed.

An example of a mica masterbatch suitable for use in the composition of the present invention is that available from Transmit Technologies Group LLC under the tradename Polymic 40PE-100 dp.

It will be apparent to the person skilled in the art that the composition of the present invention and the mica masterbatch may also comprise additional additives without deviating from the invention as claimed. These additives may include EAOs that have not been rheology modified, process oils, plasticizers, specialty additives and pigments. The specialty additives include flame retardants; antioxidants; surface tension modifiers; anti-block agents; lubricants; antimicrobial agents such as organometallics, isothiazolones, organosulfurs and mercaptans; antioxidants such as phenolics, secondary amines, phosphites and thioesters; antistatic agents such as quaternary ammonium compounds, amines, and ethoxylated, propoxylated or glycerol compounds; hydrolytic stabilizers; lubricants such as fatty acids, fatty alcohols, esters, fatty amides, metallic stearates, paraffinic and microcrystalline waxes, silicones and orthophosphoric acid esters; mold release agents such as fine-particle or powdered solids, soaps, waxes, silicones, polyglycols and complex esters such as trimethylol propane tristearate or pentaerythritol tetrastearate; pigments, dyes and colorants; plasticizers such as esters of dibasic acids (or their anhydrides) with monohydric alcohols such as o-phthalates, adipates and benzoates; heat stabilizers such as organotin mercaptides, an octyl ester of thioglycolic acid and a barium or cadmium carboxylate; ultraviolet light stabilizers such as a hindered amine, an o-hydroxy-phenylbenzotriazole, a 2-hydroxy,4-alkoxyphenone, a salicylate, a cyanoacrylate, a nickel chelate and a benzylidene malonate and oxalanilide; and zeolites, molecular sieves and other known deodorizers. A preferred hindered phenolic antioxidant is Irganox™ 1076 antioxidant, available from Ciba-Geigy Corp.

Additionally, fillers may also be useful in conjunction with the composition of the present invention. Such fillers may include carbon black, glass, metal carbonates such as calcium carbonate, metal sulfates such as calcium sulfate, talc, clay or graphite fibers. These fillers may impart further scratch and mar resistance properties to the composition.

The additional additives, if used, are typically present in an amount of less than about 45 %, based on the total composition weight. The amount is advantageously from about 0.001 to about 20 %, preferably from about 0.01 to about 15 % and more preferably from about 0.1 to about 10%.

Where mica is added as a masterbatch, both the masterbatch and the soft ethylene elastomer component may be added to the feed throat of the extruder at the same time. Alternatively, the mica masterbatch may be added directly to the extruder during mixing. Where mica is added to the soft ethylene elastomer component in powder form, the compatibilizer must be added to the soft ethylene elastomer component first or at the same time as the mica. The mica should only be added after or at least at the same time as the compatibilizer has already been added to the ethylene elastomer component.

Addition to the feed throat, will require that the feed throat be kept at a temperature cold enough to prevent bridging of components in the feed throat. Subsequent processing of the composition occurs by mixing at a temperature of from about 180 to 220°C, preferably from about 190 to about 210°C.

It will be apparent to those skilled in the art that many types of mixing equipment may be used to make the composition of the present invention including a ferrous continuous mixer, single screw or twin screw extruder and buss kneader.

The compositions of the present invention may be formed into articles of manufacture such as parts, sheets, or other forms using any one of a number of conventional procedures for processing elastomer compositions. The compositions can also be drawn into films, multi-layer laminates or extruded sheets, compounded with one or more organic or inorganic substances, injection-molded into articles such as knobs, handles for appliances or consumer goods, used for profile-extruded articles, or used for coated

fabrics or other film and sheet applications. The compositions may be useful in the manufacture of automobile interior parts, automobile exterior parts, consumer goods with soft touch grips and consumer appliances with soft touch surfaces. Other applications may also be possible and are within the knowledge of the person skilled in the art.

The compositions of the present invention have surprising improved scratch and mar resistant properties relative to other ethylene elastomer compositions.

The following example illustrates, but does not limit the present invention.

Example

All compositions used were made on a twin-screw extruder (38:1 L/D) at a processing temperature of between 190 and 210°C. The throughput was about 150 lbs/hr at a screw speed of 350 RPM. The melt was pelletized using an underwater pelletizer with the cutting water kept cold at about 11-14°C.

The compositions were formed into embossed sheets using conventional processing means. The embossed sides and "smooth" sides (non-embossed side) of each of the sheets were tested for the following properties:

- (a) shore A hardness on sheet stock under constant load (5 seconds);
- (b) taber weight loss in milligrams after 3000 cycles, 250 gm weight and CS-10 wheel on glossy side and on grained side (this is a measure of abrasive resistance);
- (c) % Gloss on smooth side at 20 deg and 60 deg angle; and,
- (d) scratching using 0.25 mm diameter indenter under 50, 100, 200, 300, 400, 500, 600 and 700 gm weight on smooth and grained sides.

Of the sheets tested, the "Control" sample is the unmodified composition—called Engage® 8957, commercially available from DuPont Dow Elastomers. This composition comprises the following components:

Engage® 8100 (DDE)	44.72 % by weight
Engage® 8842 (DDE)	29.81 % by weight

Polypropylene homopolymer (12 MFR) - Basell PDC 1274	22.97 % by weight
Vul-Cup® 20P (Geo Specialty Chemicals) 20% organic peroxide/5% silica/75% polypropylene	2.5 % by weight

The composition containing a mica masterbatch is referred to as "A". The composition of the masterbatch was: 10% functionalised resin as a coupling agent (compatibilizer), mica, Engage® 8100, processing aid, process stabilizer, heat stabilizer and 1% paraffin oil.

The mica comprised 7 % by weight of the final composition. The composition of the remaining samples B, C and D is as set out below.

Table 1: Composition of Samples

Sample	Base Compound	Additive	% wt additive in final composition
Control	Engage® 8957	None	---
A	Engage® 8957	Mica Masterbatch	7 % mica
B	Engage® 8957	Nordel® 3722P (EPDM thermoplastic rubber)	20 % EPDM thermoplastic rubber
C	Engage® 8957	Fatty amide (erucamide)	0.5 % fatty amide
D	Engage® 8957	silicone masterbatch	2 % silicone

As shown in Table 2, the mica-containing compound, namely sample A, had the best scratch and mar resistance properties. The softness of the mica-containing compound as measured by Shore A measurement was not significantly decreased with the addition of the mica masterbatch.

Table-2 Comparison of properties of extruded sheet stock

ID	Shore A Hardness 5 sec	% Gloss 60 deg Smooth Side	% Gloss 80 deg Smooth Side	Taber Wt Loss, mg 3K cycle 250 gm CS-10	Taber Wt Loss, mg 3K cycle 250 gm CS-10	Relative Scratch Rank Grained Side 0.25D mm		Relative Scratch Rank Smooth Side 0.25D mm		Relative Scratch Rank Smooth Side 0.50D mm	
						Rank Grained Side 0.25D mm	Rank Smooth Side 0.25D mm	Rank Smooth Side 0.50D mm	Rank Grained Side 0.50D mm	Rank Smooth Side 0.50D mm	Rank Grained Side 0.50D mm
Control	80A	5	24	2.3	1.4	3	4	5	5	5	5
153-04	82A	2.84	10.12	5.3	<1	1	1	1	1	1	1
153-05	80A	3.88	6.64	1	<1	4	5	4	4	3	3
110-03	82A	4.4	17.58	9	1	5	3	3	3	4	4
110-05	82A	10.54	30.86	5.3	0.3	2	2	2	2	2	2

WE CLAIM:

1. A composition comprising a soft ethylene elastomer component and an effective amount of mica and compatibilizer to impart improved scratch and mar resistance properties to the composition while not decreasing the softness of the composition.
2. The composition of claim 1, wherein the mica comprises from about 1% to about 20%, preferably from about 2% to about 15%, more preferably from about 3% to about 12% and most preferably from about 4% to about 10% by weight of the total composition.
3. The composition of claim 1 or 2, wherein the compatibilizer comprises from about 0.1% to about 7%, preferably from about 0.25% to about 5% by weight of the composition.
4. The composition of any one of claims 1 to 3, wherein the ethylene elastomer component is a rheology-modified, substantially gel-free thermoplastic elastomer composition comprising at least one elastomeric EAO polymer or EAO polymer blend and at least one high melting polymer selected from polypropylene homopolymers and propylene/ethylene copolymers, the composition having at least three of four characteristics, the characteristics being a STI \geq 20 a MS \geq 1.5 times that of the composition without rheology modification a ST \geq 10°C greater than that of the composition without rheology modification, and an UST limit \geq 10°C greater than that of the composition without rheology modification.
5. The composition of claim 4, wherein the rheology modification is peroxide induced using a level of peroxide sufficient to provide the combination of characteristics, the peroxide being an organic peroxide selected from α , α' -bis(t-butylperoxy)-diisopropylbenzene, dicymyl peroxide, 2,5-dimethyl-2,5-di-(t-butyl peroxy)hexane, di-t- butylperoxide, 2,5-di(t-amyl peroxy)-2,5-dimethylhexane, 2,5-di-(t-

butylperoxy)- 2,5-diphenylhexane, bis(alpha-methylbenzyl)peroxide, benzoyl peroxide, t-butyl perbenzoate and bis(t-butylperoxy)-diisopropylbenzene.

6. The composition of claim 5 further comprising at least one suitable co-agent with the peroxide.
7. The composition of any one of claims 1 to 6, further comprising at least one additive selected from the group consisting of EAOs that have not been rheology modified, process oils, plasticizers, specialty additives and pigments.
8. The composition of claim 6, wherein the specialty additives comprise one or more additives selected from the group consisting of: flame retardants; antioxidants; surface tension modifiers; anti-block agents; lubricants; antimicrobial agents such as organometallics, isothiazolones, organosulfurs and mercaptans; antioxidants such as phenolics, secondary amines, phosphites and thioesters; antistatic agents such as quaternary ammonium compounds, amines, and ethoxylated, propoxylated or glycerol compounds; hydrolytic stabilizers; lubricants such as fatty acids, fatty alcohols, esters, fatty amides, metallic stearates, paraffinic and microcrystalline waxes, silicones and orthophosphoric acid esters; mold release agents such as fine-particle or powdered solids, soaps, waxes, silicones, polyglycols and complex esters such as trimethylol propane tristearate or pentaerythritol tetrastearate; pigments, dyes and colorants; plasticizers such as esters of dibasic acids (or their anhydrides) with monohydric alcohols such as o-phthalates, adipates and benzoates; heat stabilizers such as organotin mercaptides, an octyl ester of thioglycolic acid and a barium or cadmium carboxylate; ultraviolet light stabilizers such as a hindered amine, an o-hydroxy-phenylbenzotriazole, a 2-hydroxy,4-alkoxyphenone, a salicylate, a cyanoacrylate, a nickel chelate and a benzylidene malonate and oxalanilide; and zeolites, molecular sieves and other known deodorizers.
9. A process for preparing the composition of any one of claims 1 to 8, comprising the steps of:

- a. preparing a mica masterbatch comprising mica, compatibilizer and base resin;
- b. mixing the mica masterbatch with a soft ethylene elastomer composition at a mixing temperature from about 180°C to about 220°C, preferably from about 190°C to about 210°C; and,
- c. pelletizing the composition at a second temperature below the mixing temperature.

10. The process of claim 9 wherein the compatibilizer is a functionalised polyolefin.

11. The process of claim 9 or 10 wherein the base resin is an ethylene elastomer.

12. The process of any one of claims 9 to 11, wherein the compatibilizer comprises from about 1 to about 20 %, preferably from about 5 to about 15% by weight of the masterbatch.

13. The process of any one of claims 9 to 12, wherein the mica comprises from about 10 to about 90%, preferably from about 30 to about 60% by weight of the masterbatch.

14. A process for preparing the composition of any one of claims 1 to 8 comprising the steps of:

- a. adding from about 1 to about 20%, preferably from about 2 to about 15%, more preferably from about 3 to about 12% and most preferably from about 4 to about 10% mica to a soft ethylene elastomer composition at a mixing temperature from about 180°C to about 220°C, preferably from about 190°C to about 210°C; and,
- b. pelletizing the composition at a second temperature below the mixing temperature.

15. The process of claim 14 wherein a compatibilizer is added to the ethylene elastomer composition in step a before or at the same time as the mica.

16. The process of claim 14 wherein the soft ethylene elastomer composition comprises a compatibilizer.
17. An article of manufacture having at least one component thereof fabricated from the composition of any one of claims 1-8, the article of manufacture is selected from the group consisting of: automobile interior parts, automobile exterior parts, consumer goods with soft touch grips and consumer appliances with soft touch surfaces.

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